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PATENT SPECIFICATION

632,247



Application Date: July 4, 1947.

No. 17747 47.

Complete Specification Accepted: Nov. 18, 1949.

Index at acceptance: - Classes 1(ii), E2a2; and 22, Filal.

COMPLETE SPECIFICATION

Improvements in or relating to Silicon Carbide Articles and Methods of Forming them

(A Communication from Trie Campontus of the carefully and existing under the Laws of the State of Delowers, United States of America, of Ningara Falls, in the County of Ningara, and State of New York, United States of Americaa).

1. Anneura Abbert, a British Subject, of 111/112, Hatton Garden, London, E.C.1, do hereby declare the nature of this

11/112, Hatton Garden, London, B.C.1,
10 do hereby declare the nature of this
invention and in what manner the same is
to be performed, to be particularly described and accertained in and by the
following statement:—

This invention relates to a novel refractory material and to the method of making it. The object of the invention is to produce a refractory composed of substantial amounts of self-bonded alicon carbide, which refractory can be made in relatively thin sections, which has a high load carrying capacity, which is highly resistant to thermal shocks, and which is highly oxidation resistant. The refractory material covered by the present invention may be used as softer tiles (i.e. thin flat slabs), bricks, and various other articles of any shape desired for use particularly in applications where a material having good strength at elevated temperatures is required.

The method of the present invention is distinguished from the prior art, in several important respects, one of which some that a relatively low firing temperature is required, and another of which is the absence of free carbon in the finished product. Because a low tamperature is used, the process results in substantial savings because of the cost of power or fuel required to fire the refractory and also because a cheaper, lower temperature film may be used in its manufacture than heretofore. Bonded to refractory materials containing a substantial imment of self-bonded silicon carbide, which are made by use of prior art methods, have been found to have relatively pior mechanical strengths at

clevated temperatures and to be prome to 50 the development of cracks as a consequence of rapid changes of temperature. On the other hand the refractory of the present invention may be made in vary thin sections where the particular application requires it, and yet will sustain high stresses throughout a long period of time at elevated temperatures. Such refractory will endure numerous rapid changes of temperature, without cracking 60 or sugging, and is also highly resistant to oxidation at high temperatures.

time at elevated temperatures. Such refraction will endure numerous rapid changes of temperature, without cracking 60 or sugging, and is also highly resistant to oxidation at high temperatures.

The method of the present invention consists, in one modification thereof, in heating an intimate mixture of finely 65 divided carbon and finely divided silicon which have been moulded and formed to the desired shape, it a relatively low temperature, namely, from approximately 1200°C. to: 1500°C. When approximately atoichiometric proportions are used and the mixture is so heated for a smitchle length of time under reducing or non-oxidising conditions the carbon and silicon react substantially completely 15 to form allicon carbide without the production of free carbon.

Thus, according to this invention, a refractoary article comprises cubic edition warhide crystals interlocked and at least 80 partially inter-diffused with each other with the absence of free carbon, with or without a granulated refractory filler material.

Such silican carbide is not the type go obtained in the silicon carbide furnace which is conventionally used in the prior art, wherein a mixture of sand and carbon is project around an elongated graphite core and is heated to an 60 extremely high temperature, as for example, from 2000°C. to 2500°C. The product resulting from such amventional method of manufacture is in the form of either large platy crystals or elongated 95 needle-like crystals of hexagonal silicon carbide. In the method of the present invention, because of the low tempera-

[Price

tures employed the product is composed.

Wholly or in part in the form of very small crystals, so small in fact, that when examined by the naked eye, they appear to be amorphous, and which upon examination, as by use of X-ray diffraction maked to be cubic silicon carbide.
The product may consist substantially

10 wholly of such cubic silicon carbide with the minute crystals closely interlocked to that the product has a very high mechanical strength. In modifications of my invention, which are employed where 15 products having properties different from those of the product above described are desired, there is employed a substantial amount of filler material which is incorporated initially with the mixture of carbon and allicon. Such filler material may be, for example, ordinary hexagonal afficing carpide of one or more meer presed shiming, mulite, kyunite, or other refrac-tory grain, or it may be a clay.

When sufficient clay is used to set as a supplementary bond, as for instance when

20 30% by weight of the product is employed, the product has a mechanical strongth and toubliness which allow it to 30 withstand especially hard use in the way

of mechanical and heat shocks.

of mechanical and heat anocis.

Two or more of such filler materials may be used together. In cases where such filler materials are used, the product so consists of such filler uniformly distributed throughout the resulting refractory shape and a bond of interlocked cubic control of the resulting restriction or hide controls. silicon carbide crystals. It is possible; by use of the filler or varying amounts of 40 clay to modify and predetermine each properties of the refractory as mechanical strength; coefficient of expansion, heat conductivity, and resistance to thermal

The cubic silicon carbide, formed as a result of the reaction between the carbon and alicon of the mixture, bonds itself and the filler material, it such is used, into a strong coherent mass by reason of the interlecting of the cubic silicon car-

bide crystals with each other and the granular filler, and by reason of the at least partial interdiffusion between adju-cent onlic silicon parhide crystals. When

55 the refractory is employed at temperatures above that at which it was formed, it is self-strengthening, since such tempera-ture will further the inter-diffusion between silicon carbide crystals. If such 60 imperature is high enough, the cubic

silicon carbide changes partially or the crystals strongly interlocked or joined. The product of the present invon-65 tion is therefore self-strengthening under

high temperature operating conditions. The process of the present invention is corried out as follows: The components of the mixture of which the refractory is to be composed, as for instance, finely divided carbon and finaly divided silicon, if no filler is to be employed, or such materials plus hexagonal silicon carbide in the desired guit size where such material is to be used as a filler, are thoroughly mixed for a long enough time to insure uniform distribution of the components throughout the mixture. arder to permit the mixture to be molded, a temporary hinder is added. Any of the usual and well known temporary hinders or resins of both dry and liquid form may be used alone or in workable combina-tions. The mix is then formed by the usual forming methods, under presences equivalent to 1000—7000 lbs. per square equivalent to 1000—7000 lbs. per square inch, after which the shapes are slowly dried in an oven at moderate temperature. After drying, the shapes are fixed in a kiln under either reducing or non-uxidizing conditions. In one method the shapes are packed in coke which, upon heating, provides a reducing atmosphere. In another method, a furnace into which is introduced either a reducing or non-uxidizing gaseous atmosphere is employed. employed.

The heating cycle employed for such fixing operation may obviously be varied considerably. Naturally, the cycle should 100 be such as to heat the shapes gradually enough to prevent their being exacted. The shapes should then be held at a temperature of from 1200 to 1500 °C., or slightly above, for a long enough period 105 to insure complete reaction between the carbon and the silicon metal. No definite figures can be given for the length of holding of the product at this tempera-ture since this obviously depends to some 110 extant upon the thickness of the cross-section of the product. As an example only, where a rolatively thin setter tile is made, it has been found satisfactory to As an example. heat the product from room temperature 115 to approximately 1260°C. in a period of twelve hours, then to slowly raise the temperature over a period of eighteen hours to about 1450° U., and to cool it back to room temperature at a rate com. 120

parable to the rate of heating.

As has been indicated above, composition of the product may be widely varied, depending upon the use for which it is intended. The ratio of the weight of 125 pilican to the weight of carbon employed in the practice of this invention should be such generally to provide for their complete reaction to form silicon carbide thus avoiding the presence of free carbon 180

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wholly of cubic silicon carbide, with no appreciable excess of either carbon or silicon. Without the use of such atmospheres there occur relatively large losses of silicon by vaporization and losses of carbon by oxidation especially from the outer portions of the product; when this takes place the composition of the product is not uniform and is not subject to the rigid control made possible by the prac-

Although silicon has been described as being used in that form, it is obvious that the allicon may be derived from finely 15 comminuted alloy which is employed in the mix instead of silicon. Such alloy may be, for example, an aluminium silicon alloy or a ferro-silicon having a high silicon content, for example, 90%.

20 In the examples given above the carbon is added to the mixture in the form of lamp black. It is to be understood that carbon in other finely divided forms may be supplied in the present.

lamp black. It is to be understood that carbon in other finely divided forms may be employed in the process. Thus, for unstance, powdered retort carbon or powdered coke may be employed in the practice of the invention.

Having now particularly described and ascertained the nature of the said invention and in what manner the same is to be performed, as communicated to me by my foreign correspondents, declare that what I claim is:—

1. A refractory article comprising public 35 silicon earbide mystels interlocked and at least partially inter-diffused with each other with the absence of free carbon, with or without a granulated refractory filler material.

ther material.

2. A refractory article according to claim I, and comprising silicon carbide grain of heragonal crystal habit interlocked with the cubic efficient carbide crystals, and distributed throughout the stricle in a substantially uniform manner.

3. A refractory article according to claim 2, wherein the silicon carbide gmin of hexagonal crystal habit is present in

up to 90% by weight of the article.

4. A refractory article according to any
of the preceding claims, wherein there is
also present elemental silicon

5. A refractory article according to claim 4, wherein the silicon plus the cubic 55 silicon curbide commises from 20% to substantially 100% by weight of the article, the elemental silicon being present in amounts up to 16.65% by

weight of the article.

6. The process of manufacturing refractory articles comprising forming a mix of substantial amounts of powdered silicon and finely divided carbon, the ratio of the weight of the silicon and the weight of finely divided carbon in the mix lying between 2.34 and 3, mixing the components thoroughly with a temperary binder so that the mixture is rendered workable, forming the mixture to shape, drying the shape and firing in a non-oxidizing or reducing atmosphere at a temperature of at least 1200° C, for such a period of time that a substantial portion of the silicon reacts with the finely divided carbon to form cubic silicon carbide crystals which are so interlocked with each other that a mechanically strong shape results.

when each other that a mechanically strong shape results.

7. A process of manufacturing refractory articles according to claim. 6, wherein the firing of the formed mixture is carried out within a temperature range of 1200° C. to 1500° C.

68. A process of manufacturing refrace 55 tmy articles according to claim 6 or claim 7, wherein there is incorporated in the initial mix a refractory filler material.

9. A process of manufacturing refrac- at tary articles according to claim 8, wherein said refractory filler material comprises silicon carbide grain.

silicon carbide grain.
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